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Multi-Media, Multi-Concentration Dioxin and Furan Analytical Service for Superfund (DLM02.0)

Office of Superfund Remediation and Technology Innovation (OSRTI)
Analytical Services Branch (ASB) (5102G)

Quick Reference Fact Sheet

Under the legislative authority granted to the U.S. Environmental Protection Agency (EPA) under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA), EPA develops standardized analytical methods for the measurement of various pollutants in environmental samples from known or suspected hazardous waste sites. Among the pollutants that are of concern to the EPA at such sites are a series of chlorinated dibenzo-p-dioxins (CDDs) and chlorinated dibenzofurans (CDFs) that are analyzed using High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS). The Analytical Services Branch (ASB) of EPA's Office of Superfund Remediation and Technology Innovation (OSRTI) offers an analytical service that provides data from the analysis of water, soil, sediment, sludge, non-human tissue, ash, oil, and oily matrices for use in the Superfund decision-making process. Through a series of standardized procedures and a strict chain-of-custody, the dioxin and furan analytical service produces data of known and documented quality.

DESCRIPTION OF SERVICES

The dioxin and furan analytical service provides a flexible contractual framework for laboratories to apply EPA analytical methods for the isolation, detection, and quantitative measurement of seventeen 2,3,7,8-substituted tetra- through octa- CDDs/CDFs in water, soil, sediment, sludge, non-human tissue, ash, oil, and oily matrices. EPA ASB has prequalified laboratories that use the Dioxin and Furan Statement of Work (SOW) DLM02.0 to provide this service. Data evaluation can be performed by the data requestor using the National Functional Guidelines (NFG) document provided by EPA ASB. The standard data Turnaround Time (TAT) for this service is 35 days after laboratory receipt of the last sample in the Sample Delivery Group (SDG). This TAT can be changed to meet project-specific requirements.

REQUESTING THIS FLEXIBLE SERVICE

The dioxin and furan analytical service can be requested by EPA Regions and other interested parties by submitting a Task Order (TO) to EPA ASB. These TOs can modify the SOW to meet project-specific requirements [e.g., changes in TAT, detection limits, or the Target Compound List (TCL)]. The DLM02.0 SOW and the NFG document can be accessed at: http://www.epa.gov/superfund/programs/clp/dlm2.htm

DATA USES

The dioxin and furan analytical service provides data that EPA uses for a variety of purposes such as: determining the nature and extent of contamination at a hazardous waste site; assessing priorities for response based on risks to human health and the environment; determining appropriate clean-up actions; and determining when remedial actions are complete. The data may be used in all stages in the investigation of hazardous waste sites, including: site inspections; Hazard Ranking System (HRS) scoring; remedial investigation/feasibility studies; remedial design; treatability studies; and removal actions. In addition, this service provides data that are available for use in Superfund enforcement/litigation activities.

TARGET COMPOUNDS

The applicable target compounds and Contract Required Quantitation Limits (CRQLs) for this service are listed in **Table 1**. For water samples, the lowest reportable CRQL is 10 pg/L. For solid samples, the lowest reportable CRQL is 1.0 ng/kg. The specific CRQLs are highly matrix-dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

Table 1. Target Compound List (TCL) and CRQLs

CDD/CDF	Water (pg/L)	Solids (ng/kg)
2378-TCDD	10	1.0
12378-PeCDD	50	5.0
123678-HxCDD	50	5.0
123478-HxCDD	50	5.0
123789-HxCDD	50	5.0
1234678-HpCDD	50	5.0
OCDD	100	10
2378-TCDF	10	1.0
12378-PeCDF	50	5.0
23478-PeCDF	50	5.0
123678-HxCDF	50	5.0
123789-HxCDF	50	5.0
123478-HxCDF	50	5.0
234678-HxCDF	50	5.0
1234678-HpCDF	50	5.0
1234789-HpCDF	50	5.0
OCDF	100	10

METHODS AND INSTRUMENTATION

For water samples, the stable isotopically-labeled analogs for fifteen of the 2,3,7,8-substituted CDDs/CDFs are spiked into a 1 L sample.

Water samples with no visible particles are extracted with methylene chloride using a separatory funnel or are vacuum-filtered through a glass-fiber filter on top of a solid-phase extraction (SPE) disk. The extract is concentrated for cleanup.

Water samples containing visible particles are vacuum-filtered through a glass-fiber filter. The particles and filter are extracted in a Soxhlet/Dean-Stark (SDS) extractor and the filtrate is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is concentrated and combined with the SDS extract prior to cleanup.

For soil/sediment samples, the labeled compounds are spiked into a sample containing 10 g (dry weight) of soil/sediments. Samples containing coarse soil/sediments are ground or homogenized. The soil/sediments are then extracted using an SDS extractor.

For fish and other tissue, a 20 g aliquot of frozen or non-frozen sample is homogenized and a 10 g aliquot is spiked with the labeled compounds. The frozen sample is mixed with sodium sulfate, allowed to dry overnight, and extracted for 12-24 hours using methylene chloride:hexane (1:1) in an SDS extractor. The non-frozen sample is allowed to equilibrate, then 200 mL hydrochloric acid and 200 mL methylene chloride:hexane (1:1) are added and the bottle is agitated for 12-24 hours. In both cases, the extract is evaporated to dryness and the lipid content is determined.

For all samples, the extracts are cleaned and injected with two internal standards; one to determine Percent Recoveries (%R) of tetra- and penta- CDD/CDF congeners and the other to determine the recoveries of hexa-, hepta-, and octa- CDD/CDF congeners. An aliquot of the extract is injected into the HRGC for separation and the analytes are detected by a HRMS. **Table 2** summarizes the methods and instruments used in this analytical service.

DATA DELIVERABLES

Data deliverables for the dioxin and furan analytical service include the hardcopy data reporting forms and supporting raw data. Certain TOs may require the use of the Electronic Data Deliverable (EDD) format. The EDD reporting requirements will be outlined in the TO. The laboratory must submit data to EPA within 35 days after laboratory receipt of the last sample in the SDG, or as stated in the TO. The EPA Regions then review the data, based on project-specific requirements and the NFG document.

Table 2. Methods and Instruments

Matrix	Preparation Method	Analytical Instrument
Water (no visible particles)	SPE or a separatory funnel with methylene chloride for extraction.	HRGC/HRMS analysis
Water (visible particles)	Vacuum filtration with SDS extractor for particles and filter extraction. Use a separatory funnel for filtrate extraction with methylene chloride.	HRGC/HRMS analysis
Soil/Sediment	SDS extraction.	HRGC/HRMS analysis
Fish and Other Tissue (frozen)	Mix with sodium sulfate and allow to dry. Extract with methylene chloride:hexane (1:1) using an SDS extractor.	HRGC/HRMS analysis
Fish and Other Tissue (non-frozen)	Mix with equal volumes of hydrochloric acid and methylene chloride:hexane (1:1). Agitate for 12-24 hours.	HRGC/HRMS analysis

QUALITY ASSURANCE (QA)

The QA process consists of management review and oversight at the planning, implementation, and completion stages of the environmental data collection activity. This process ensures that the data provided are of the quality required.

During the *planning* of the data collection program, QA activities focus on defining data quality criteria and designing a Quality Control (QC) system to measure the quality of data being generated. During the *implementation* of the data collection effort, QA activities ensure that the QC system is functioning effectively, and the deficiencies uncovered by the QC system are corrected.

After environmental data are collected, QA activities focus on assessing the quality of data to determine its suitability to support enforcement or remedial decisions.

Each contract laboratory prepares a Quality Assurance Plan (QAP) with the objective of providing sound analytical chemical measurements. The QAP must specify the policies, organization, objectives, and functional guidelines, as well as the QA/QC activities designed to achieve the data quality requirements for this analytical service.

QUALITY CONTROL (QC)

The QC process includes those activities required during analytical data collection to produce data of known and documented quality. The analytical data acquired from QC procedures are used to estimate and evaluate the analytical results and to determine the necessity for, or the effect of, corrective action procedures. The QC procedures required for this analysis are shown in **Table 3**. A number of optional cleanup procedures are also available in this SOW.

PERFORMANCE MONITORING ACTIVITIES

Laboratory performance monitoring activities are provided primarily by ASB and the Regions to ensure that contract laboratories are producing data of the appropriate quality. EPA performs on-site laboratory evaluations, electronic data audits, data package audits, HRGC/HRMS tape audits, and evaluates laboratory performance through the use of blind Performance Evaluation (PE) samples.

CONTACTING USEPA

For more information, or to submit suggestions to improve this analytical service, please contact:

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Table 3. Quality Control (QC)

QC Operation	Frequency
ANALYSES	
HRMS System Tune	Every 12 hours prior to analysis of calibration standards, samples, blanks, and QC samples, and at the end of each 12-hour shift or analytical sequence.
Window Defining Mixture (WDM) **NOTE:** The WDM and Isomer Specificity Check (see below) solutions may be combined into a single solution [Column Performance Solution (CPS)], provided that the combined solution contains the isomers needed to determine that the criteria for analysis are met.	Every 12 hours for each instrument used for analysis and whenever adjustments or instrument maintenance activities are performed that may affect Retention Times (RTs). Precedes Initial Calibration and Calibration Verification; follows the HRMS System Tune.
Isomer Specificity Check **NOTE:** The WDM and Isomer Specificity Check solutions may be combined into a single solution (CPS), provided that the combined solution contains the isomers needed to determine that the criteria for analysis are met.	Every 12 hours for each instrument used for analysis and whenever adjustments or instrument maintenance activities are performed that may affect RTs. Performed after or simultaneously with the WDM and before any Initial Calibration.
Initial Calibration	Upon contract award, upon initial setup of instruments, prior to analysis of samples and required blanks, whenever any corrective action is taken that may change or affect the initial calibration criteria, and each time Calibration Verification fails to meet the technical acceptance criteria.
Calibration Verification [Mid-Point Calibration Standard (CS3) Relative Response (RR) and Relative Response Factor (RRF)]	Prior to the beginning of every 12-hour period during which sample data are collected, but following each injection of Column Performance Solution (CPS), and at the end of each 12-hour period or analytical sequence.
STANDARDS	
Internal Standards	Added to all extracts prior to analysis.
Cleanup Standard	Added to all extracts prior to cleanup.
BLANKS	
Performance Evaluation (PE) Samples	Prepared and analyzed (if provided) with each set of 20 field samples.
Laboratory Control Sample (LCS)	Prepared and analyzed with each group of 20 field samples or less of a similar matrix in an SDG. LCS analysis precedes analysis of samples from the same SDG.
Method Blank	Prepared and analyzed with each group of 20 field samples or less, or each time samples are extracted to determine the level of contamination associated with the processing and analysis of samples. Method Blank analysis precedes analysis of samples from the same SDG.
INSTRUMENT CALIBRATION	
Gel Permeation Chromatography (GPC) Calibration (optional)	Upon contract award, upon initial setup of instruments, when the Gas Chromatography (GC) column is changed, when channeling occurs, and once every 7 days when samples are cleaned using GPC.